SHARED INSTRUMENTATION FACILITY

Colorado School of Mines’ Shared Instrumentation Facility houses a wide range of state of the art instrumentation supporting research in Electron & Scanning Probe Microscopy, Mass Spectrometry, X-ray Diffraction & Computed Tomography, Cleanroom & Thin Film Processing, Rock & Fluid Multiphysics, and X-Ray Photoelectron Spectroscopy. Details regarding selected equipment available at Mines for the proposed work are provided below.

CLEANROOM AND THIN FILM PROCESSING

CLASS 1000 CLEANROOMS EQUIPMENT
Mines’ Shared Instrumentation Facility houses two Class 1,000 Cleanrooms with photolithography capabilities for substrates up to 3 inch (76 mm) diameter and features down to 2 μm resolution and encompass the equipment outlined below.

HIGH TEMPERATURE THERMAL EVAPORATOR
In thermal evaporation, a source material is evaporated in a vacuum through the application of a large applied current. The vacuum allows vapor particles to travel directly to the substrate, where they condense back to a solid state. The Large thermal evaporator has 4 material holders which can be used for a variety of evaporable materials, and a large substrate mount that can hold 16 3”x3” substrates.

UV MASK ALIGNER
Karl Suss MJB3 UV400 Mask Aligner is an instrument enabling photolithography, which is a microfabrication process used to selectively remove parts of a thin film to create a pattern or a design onto a substrate by means of UV lamp. It fits substrates up to 6” x 6” and exposes with a UV400nm source at 250W.

RAPID THERMAL ANNEALER
The ULVAC-RIKO MILO-5000 mini lamp annealer provides rapid heating of a wafer from ambient temperature to a desired temperature in controlled gas environments. Once the wafer reaches this temperature, it is held there for a few seconds and then cooled. Operational temperature ranges from room temperature to 1200 °C. Sample size: 20 x t2 (mm)

PLASMA PREEN
The Plasma-Preen System is a plasma cleaning/etching system with O2 and Ar capabilities which uses microwaves to excite the plasma.

SPIN COATER/HOT PLATE
Spin coating is a procedure used to deposit solution processed materials into uniform thin films onto flat substrates. Film thickness can be controlled by varying spin speed from 100 – 10,000 RPM.

FUME HOODS
Multiple ventilated work areas for sample preparation with high airflow designed to limit exposure to hazardous or toxic fumes, vapors or dusts.
CLASS 10,000 CLEANROOM EQUIPMENT

Mines’ Shared Instrumentation Facility houses one Class 10,000 Cleanroom Lab houses processing equipment for making and characterizing electronic devices and encompasses the equipment outlined below.

DIFFUSION FURNACE
Tube furnace used in the manufacturing of semiconductor components. Used to add doping impurities into high purity silicon wafers, thereby creating embedded semiconductor devices.

DRY OXIDATION FURNACE
Furnace used for growing dry oxides.

WET OXIDATION FURNACE
Wet oxidation uses clean steam. The H2O molecule is smaller than the O2 molecule and diffuses through the silicon dioxide layer faster. This allows for the growth of thicker films.

VACUUM FURNACE
A vacuum furnace is a type of furnace in which the product in the furnace is surrounded by a vacuum during processing. The absence of air or other gases prevents oxidation and heat loss from the product through convection, and removes a source of contamination.

REACTIVE ION ETCHER
The AutoGlow 200 reactive ion etcher is a table-top plasma system that can be used for plasma cleaning or RIE processing with O2 and SF6 plasmas. It can process as low as 10 W, or as high as 300 W in one-watt increments. The AutoGlow 200 is suitable for lab, failure analysis, or production applications. It can perform a host of applications, including cleaning, removing photoresist, prebonding, organic removal, activation and plasma etching.

REACTIVE ION ETCHER
The March CS 1701 Reactive Ion Etcher delivers performance often associated with high-investment etching tools. The system is excellent for metal etching, silicide etching, and etching of III-V compounds, anisotropic etching of nitrides, oxides and polyimides.

Key performance features of the CS 1701 system include the large DC bias and the ability to control process pressure independent of gas flow. The system allows users a wide variety of etch profiles ranging from anisotropic requiring high aspect ratios to sloped walls. The system has 3 process gases (O2, Ar, SF6) and a N2 purge.

RAPID THERMAL ANNEALER
The ULVAC-RIKO MILA-5000 mini lamp annealer provides rapid heating of a wafer from ambient temperature to approximately desired temperature in controlled gas environments. As soon as the wafer reaches this temperature, it is held there for a few seconds and then cooled. Temperature range: room temperature to about 1200 °C. Sample size: 20 x t2 (mm).

FUME HOODS
A fume hood is a type of local ventilation device that is designed to limit exposure to hazardous or toxic fumes, vapors or dusts.
FOUR POINT PROBE
An apparatus for rapidly measuring the resistivity of thin film samples. By passing a current through two outer probes and measuring the voltage through the inner probes, one can easily measure substrate resistivity.

HALL SYSTEM
The HL5500PC is a high-performance Hall Effect Measurement System. It enables measurement of resistivity, carrier concentration and mobility on a wide range of semiconductors and with minimum effort in sample preparation. The HL5500PC is designed as a modular system and the basic instrument can measure sheet resistivity’s up to a few MOhm/square and Hall voltages of a few µV. Samples can have Van der Pauw, bar or bridge shaped geometries, and dual temperature measurements (at room temperature and 77 K) are readily achievable.

- Thickness should be less than 1/15 of the peripheral length (less than 1mm), and uniform to ±1%.
- Sheet resistivity’s of 0.1 mΩ/square to in excess of 1 MΩ/square
- Optional to extend sheet resistivity measurements to 10¹¹Ω/square
- Max Measurement Diameter: 25 mm
- Magnet: Permanent with field reversal by magnet rotation
- Support for van der Pauw, Hall bar and bridge samples
- Integration, delay & repeat measurement modes increase flexibility and accuracy on difficult to measure samples
- Easy to export data and information

ELECTRICAL PROBE STATION
The probe station measures the electrical properties of semiconductor samples under an optical microscope. With 4 micropositioner probes and an adjustable connection to a Keithley sourcemeter, a curve tracer, and a capacitance bridge, one can measure the electrical properties of microfabricated devices

MATERIALS CHARACTERIZATION LAB EQUIPMENT
Mines’ Shared Instrumentation Facility Materials Characterization Lab houses several tools for the surface, morphological, optical, and cryogenic characterization of materials and encompasses the equipment outlined below.

DIGITAL INSTRUMENTS AFM
The Digital Instruments Atomic Force Microscope is a high-resolution scanning probe microscope with sub-nanometer vertical resolution and sub-micron horizontal resolution. The system uses a sharp tip on the edge of a cantilever to scan a samples surface and record fluctuations. It can be used to characterize surface roughness, grain boundaries, lithographic structures, and polymeric materials.

PHENOM SEM
The Phenom Scanning Electron Microscope is a tabletop SEM which features combined optical- electron images for easy sample navigation. It is designed for ease of use and rapid sample loading. It can achieve up to 100,000 X magnification with resolution down to 15nm.

PROFILOMETER
The D-600 stylus profilometer is an instrument used to measure a surface’s profile to quantify its roughness
and step heights. The D-600 profilometer can measure fluctuations in surface profile from 1200um down to the nanometer scale.

**LOW TEMPERATURE CRYOSTAT**
The Janis cryostat is a versatile sample cooling system with an optical window and electrical contacts for electrical transport measurements. It can cover a temperature range of 4K – 300K, with a rapid 2-hour cooldown time and is connected to a Keithley digital source meter, nanovoltmeter and signal amplifier.

**UV-VIS SPECTROMETER**
The CARY 5G is a high-performance UV-VIS and NIR spectrophotometer, with a 175-3300 nm range. Ideal for measuring the absorption profiles of thin film materials.

**OPTICAL MICROSCOPE**
The lab also contains an optical microscope with backlighting and 5X- 100X magnification.

**THIN FILM DEPOSITION LAB EQUIPMENT**
Mines’ Shared Instrumentation Facility Thin Film Deposition Facility contains several tools for the creation of metallic and ceramic thin film devices and encompasses the equipment outlined below.

**ANGSTROM THERMAL EVAPORATOR**
The Angstrom thermal evaporator is a thermal evaporation system dedicated to the evaporation of metals (gold, silver, aluminum, and copper.) It has an automated deposition rate controller for ease of use and the creation of consistent reproducible films.

**SEMICORE SPUTTERING SYSTEM**
The Semicore is a 3-gun magnetron DC sputtering system with a sputter-down configuration for the deposition of high melting point metals. The system has the capability to do subsequent layered deposition of multiple metals.

**AJA SPUTTERING SYSTEM**
The AJA is a 4-gun magnetron sputtering system with a sputter-up configuration. The system has the capability to perform RF sputtering deposition for the deposition of ceramic materials, is configured to deposit magnetic materials, and can sputter multiple materials simultaneously.

**TABLETOP GOLD SPUTTER COATER**
The Denton nanocoater is a rapid deposition sputtering system configured to deposit a 20nm gold coating on samples in order to prevent surface oxidation or to provide surface conductivity for e-beam measurements.

**WIRE BONDER**
The TPT hb05 is a benchtop manual wire bonding system with gold, silver, aluminum or copper wire and a 6:1 mouse ratio. Ideal for attaching gold contact wires to microscopic features for electrical measurement.
ELECTRON & SCANNING PROBE MICROSCOPY EQUIPMENT

SPECIMEN PREP CAPABILITIES IN THE ELECTRON MICROSCOPY LAB

**GENERAL**
- Slow Speed Diamond Saws
- Grinding Wheels
- EVACTRON CombiClean Decontaminator

**SEM**
- Vibromet Polisher
- JEOL IB-0910CP Cross-Section Polisher
- Hummer IV Sputtering System (Au/C Coater)

**TEM**
- Gatan Grinding Fixture
- 3mm Foil Punch
- South Bay Model 350 3mm TEM Disk Cutter
- Gatan Dimpler
- Gatan Model 600 Dual Mill
- Fischione Electropolishing System

EQUIPMENT SPECIFIC DETAILS

**ASYLUM MFP-3D SCANNING PROBE MICROSCOPE**

The Asylum MFP-3D atomic force microscope is coupled to an optical microscope (Olympus) for simultaneous imaging and physical properties measurements at the nanoscale for a wide range of advanced materials in renewable energy and life sciences. We can measure electrical and mechanical properties of materials in direct registry with topography at nanoscale resolution under their designed operating conditions (e.g. illumination of photovoltaics and hydrated conditions for biological). The measurement of interfacial physical properties at small length scales is essential to the development of the next generation of materials for renewable energy and life sciences. Specific advanced capabilities beyond standard SPM modes (contact, tapping, phase) include:

- Optical Microscopy: The AFM head is integrated with an inverted optical microscope, facilitating the quick identification of the specific region of interest and enables optical stimulation to obtain properties such as a photoconductivity or fluorescence in registry with topology.

- Electro/Mechanical Measurements: The instrument enables spatially resolved measurements of critical material properties such as conductivity, piezoelectricity, Kelvin Probe, modulus, and viscoelasticity.

- Environmental Control: Samples may be evaluated under controlled conditions: temperature (−35°C to 300°C), humidity, fluid cells, and illumination intensity/wavelength.

- We gratefully acknowledge the National Science Foundation and Colorado School of Mines for funding this facility through award [#CBET-1532179](#CBET-1532179).
FEI HELIOS NANOLAB 600I FIB/SEM
The Helios NanoLab 600i is a SEM/FIB DualBeam workstation with a Ga ion column for imaging and sample milling and Pt deposition (GIS) capability. Nanoscale chemical analysis may be performed with an EDAX energy dispersive X-ray spectroscopy (EDS) system. The instrument is also outfitted with an electron backscattered diffraction (EBSD) system, which allows for crystallographic determination on the nanoscale, and, along with imaging capabilities, 3-D reconstructions of the material composition and crystallography. In situ manipulation for TEM foil extraction is performed using an Omniprobe Autoprobe 200 nano-manipulator, with 10 nm positioning resolution.

- Schottky Field Emission Cathode
- Resolution: 0.9 nm at 15 kV (at eucentric WD); 1.4 nm at 1 kV (at optimum WD)
- Ga Source Focused Ion Beam (FIB) – 0.5 kV to 30 kV
- Platinum Deposition (GIS) System
- Secondary (SEI) and Backscatter (BEI) Imaging
- In-lens SE detector (TLD-SE)
- In-lens BSE detector (TLD-BSE)
- Everhardt-Thornley SE
- Solid-state BF and DF STEM detector
- AutoFIB, AutoTEM, Slice and View, and EBS3 Software
- EDAX “Octane Super” SDD Energy Dispersive Spectroscopy of X-rays (EDS)
- EDAX “Hikari Super” 1400pps Electron Backscatter Diffraction (EBSD) detector
- EDAX TEAM software
- Omniprobe Autoprobe™ 200 Nano-manipulator

FEI QUANTA 600I ENVIRONMENTAL SEM
The Quanta SEM has operating vacuum modes to deal with different types of samples. High Vacuum (HiVac) is the conventional operating mode associated with all scanning electron microscopes. The two other application modes are Low Vacuum (LowVac) and ESEM. In these modes the specimen chamber is at a pressure range of 0.1 to 30 Torr (15 to 4000 Pa). Either mode can use water vapor from a built-in water reservoir, or auxiliary gas supplied by the user. Observation of outgassing or highly charging materials can be made using one of these modes without the need to metal coat the sample.

- Tungsten Cathode
- Secondary (SEI) and Backscatter (BEI) Imaging
- Three modes of operation:
  1. High Vacuum (10⁻⁶ torr)
  2. Low vacuum (0.1 – 1.0 torr)
  3. ESEM (1.0 – 10 torr)
- EDAX “Element” SDD Energy Dispersive Spectroscopy of X-rays (EDS)
- Large Sample Chamber
- Hot Stages (rated to 1500°C)
- Peltier Cold Stage
FEI TALOS F200X TEM/STEM
The FEI Co. Talos F200X 200keV field emission scanning / transmission electron microscope is located in the CoorsTek Building (Room 001P). The FEI X-FEG high brightness electron source delivers high total current — up to five times the beam current of a standard Schottky FEG — while the integrated EDS system with four silicon drift detectors (SDDs) offers mapping capabilities of up to $10^5$ spectra/sec. It has a TEM information limit better than 0.12 nm. The STEM probe allows for an imaging resolution of 0.16 nm. STEM images can be viewed in bright field or with multiple dark field detectors, including a high-angle annular dark field detector (HAADF) allowing for Z-contrast imaging.

- Schottky Field Emission Gun
- 200kV Accelerating Voltage
- Conventional and Scanning Modes (CTEM/STEM)
- High Resolution (HREM): 0.12 nm Information Limit
- Super-X EDS for Spectral Imaging
- High Angle Annular (HAADF) and Centered (CDF) Dark Field Modes

FEI TECNAI T12
Tecnai™ G2 Spirit TWIN (T12)
- Accelerating Voltage: 20-120KV
- Gun Type: W Filament/LaB6
- Line resolution (nm): 0.2
- Cs objective (mm): 2.2
- Cc objective (mm): 2.2
- Focal length (mm): 2.8
- Minimum focus step (nm): 3.0
- Maximum eucentric tilt: ± 70°
- Magnification: 18 – 650,000
- Specimen Exchange Time: < 30 seconds
- High tension switching Time: < 1 minute
- X, Y movement: 2 mm
- Elemental Analysis: EDAX Element EDS System
- Image Capture: CD Camera and Film
- Specimen Holders: Single and Double Tilt

JEOL JSF-7000F FIELD EMISSION SEM
The JSM-7000F is a field-emission scanning electron microscope with a Schottky type field-emission gun for the electron source and state-of-the-art computer technology for the image-display system. The instrument is equipped for energy dispersive X-ray spectroscopy (EDS) and electron backscatter diffraction (EBSD) analysis.

- Schottky Field Emission Cathode
- High Resolution (1.2nm @30kV)
- Secondary (SEI) and Backscatter (BEI-TOPO/COMPO) Imaging
- EDAX “Octane Pro” SDD EDS
- EDAX “Hikari Pro” 600pps EBSD detector
- Electron Lithography
**TESCAN S8252G RAMAN-SEM/FIB**
The TESCAN S8000G is an ultra-high resolution variable pressure Schottky field emission scanning electron microscope/Ga+ focused ion beam instrument for imaging and sample milling and W, Fe, and Co deposition (GIS) capabilities. It is equipped with WITec in situ confocal Raman imaging at 532 nm and 785 nm excitation wavelengths using a 100x objective, NA = 0.75. The SEM/FIB has an Everhart-Thornley detector, two retractable backscatter detectors (one water-cooled), an in-beam multi-detector, in-beam axial detector, and secondary ion detector. The instrument is also capable of 3-D imaging using any of the detectors.

Nanoscale compositional analysis may be performed with an EDAX Octane Elect Plus (30mm²) energy dispersive X-ray spectroscopy (EDS) system with APEX software.

In situ manipulation for TEM foil extraction is performed using a SmarAct nano-manipulator, with 1 nm positioning resolution and an electron flood gun for charge neutralization during ion beam milling.

The Gatan Murano heating stage allows for in situ analysis from room temperature to 950°C.

- SEM Resolution: 0.9 nm at 15 kV (at 1 mm WD)
- Ga Source Focused Ion Beam (FIB) – 0.5 kV to 30 kV, up to 100nA
- Optical resolution: diffraction limited lateral typ. 430 nm @ 532 nm excitation wavelength
- 532 nm, 75 mW diode-pumped solid state laser
- 785 nm, 125 mW diode laser
- Fully motorized, heavy-duty compucentric 5-axis cradle stage (X=256mm, Y=152mm, Z=52mm, Rotation=360°, Bidirectional tilt = −3° to 70°, 8kg weight capacity

This instrument was acquired through the support of the National Science Foundation (DMR-1828454)

**WITEC LASER CONFOCAL RAMAN MICROSCOPE**
This instrument is a WiTec Alpha300 laser confocal microscope equipped with a Raman spectrometer.
MASS SPECTROMETRY EQUIPMENT

IONTOF TOF-SIMS.V
Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) is a highly surface-sensitive analytical technique used to obtain elemental, isotopic, and molecular information from the surface of solid materials and compacted powders. This TOF-SIMS instrument features a Primary Ion Beam operating at 30 keV with a three-lens BiMn cluster nanoprobe. For sputtering sources (Secondary Ion Beams) the TOF-SIMS can utilize either a Thermal ionization Cesium source, an Oxygen electron impact gas ion source, or a fully integrated gas cluster ion source. The Bi Nanoprobe source provides high analysis currents of up to 20 pA for trace detection spectrometry and high-end depth profiling.

Each point of impact on the sample from the primary ion beam contains the entire mass spectrum as well as the X, Y and Z coordinates of that point of impact. With this information, we can create detailed ion images of the distribution of any species of interest on our sample, both in 2D and in 3D (in depth profile mode).

- High sensitivity with the ability to detect species in the parts-per-million (ppm) to parts-per-billion (ppb) range
- High mass resolution of ~0.00x amu
- Capable of ~80nm spatial resolution
- Can obtain elemental and molecular information from a mass range of 0 to 10,000+ amu simultaneously
- Depth profiling with parallel ion detection
- Species mapping in both 2D and 3D
- Ability to analyze insulators and conductors
- Retrospective analysis

ADDITIONAL FEATURES
- **Argon Gas Cluster Source** provides the ability to detect high mass polymers and depth profile through complex organic materials.
- **In-situ Focused Ion Beam** can analyze extremely rough samples, samples with voids or samples that exhibit strong local variations in density.
- **Extended Dynamic Range Analyzer** extends dynamic range up to seven orders of magnitude, allowing for simultaneous detection of normally saturated matrix species and trace species.
- **Hermetically Sealed Transfer Vessel** ability to analyze atmospheric or moisture-sensitive samples.

SCIEX 5500 TRIPLE QUAD
Liquid Chromatography-Mass Spectrometry is an analytical technique for determining an ion’s mass-to-charge ratio by partitioning the particles within a liquid and measuring the time it takes for each particle to travel through a selected mobile phase. Ultimately this helps in determining the contents of a liquid sample and can be used to analyze biochemical, organic and inorganic compounds.

A Triple Quadrupole Mass Spectrometer is a highly sensitive instrument for detecting and quantifying known compounds within a sample. Within the instrument there are three quadrupoles arranged in a series where the first quadrupole acts as a mass filter, the second functions as a collision cell wherein the ions interact with a gas, and the third quadrupole helps to detect the mass range. The ions are then fired towards a detector which analyzes the individual fragments.
SCIEX X500R QTOF

Liquid Chromatography-Mass Spectrometry is an analytical technique for determining an ion’s mass-to-charge ratio by partitioning the particles within a liquid and measuring the time it takes for each particle to travel through a selected mobile phase. Ultimately this helps in determining the contents of a liquid sample and can be used to analyze biochemical, organic and inorganic compounds.

A Quadrupole Time-of-Flight Mass Spectrometer is a very sensitive system which can be very beneficial in non-target analysis involving screening a sample for a multitude of unknown compounds. Similar to a Triple Quad MS, there are three quadrupoles arranged in a series. A QToF MS switches out the third quadrupole for a TOF mass analyzer, allowing for greater range of ion detection and mass accuracy. Using a QToF system makes it possible to perform a full ion scan to allow for future analysis of unknown compounds without needing to reacquire data.
ROCK & FLUID MULTIPHYSICS

**ADSORPTION LAB**

**Micromeritics ASAP 2020**

This Micromeritics ASAP 2020 is a gas adsorption instrument designed for core samples with very small pore sizes that range between 3 to 200 nanometers. It is mostly useful to measure the pore sizes of unconventional reservoir rocks. It has two outgassing and one analysis ports. Depending on the pore sizes, each sample measurement can take from a few hours (only surface area) to a few days (nanometer-sized pores in organic matter). The samples are measured in their powder form, and the gases that can be used for this instrument are Argon, N2, Hexane, CO2, and Water Vapor. The samples are first outgassed for 24 hours at 200 °C under vacuum condition while flowing inert gas through the sample to remove any contaminant gas. Then, the sample is subjected to increasing partial pressures to obtain a full isotherm. The following is a list of parameters which can be measured using ASAP 2020:

- Specific Surface Area
- Isotherm – Adsorption & Desorption
- Pore Size Distribution
- Total Pore Volume
- Average Pore Size

**Quadrasorb EVO/SI**

The Quadrasorb EVO/SI is a versatile gas adsorption instrument designed for high throughput. With four independent analysis stations it is capable of analyzing four samples simultaneously. Quadrasorb EVO/SI is available in a KR/MP configuration, which is equipped with a turbomolecular pump and 10-torr or 1-torr pressure transducer for measuring low surface area samples with krypton and microporous samples with nitrogen or argon. The following is a partial list of important parameters which can be determined from Quadrasorb Adsorption experiments:

- Specific Surface Area
- Isotherm – Adsorption & Desorption
- Pore Size Distribution
- Total Pore Volume
- Average Pore Size

Fluvacdegasser is used in conjunction with the QUADRASORB EVO/ SI for outgassing at vacuum condition purposes.

**Acoustic Scanning Microscope**

**FLUIDS LAB**

The Fluids Lab is a part of the Center for Rock & Fluids Multiphysics. Instrumentation in the fluids lab enables:

- Ultrasonic velocity measurements
- Electrical experiments
- Measuring effects of fluids, confining pressures, and temperatures
- Multiphysics/joint measurements
- Tools include:
Anisotropic Acoustic–Electrical Joint Measurement System

We use the system to characterize the geophysical rock properties and anisotropy, and to determine the effect of pressure and fluids on reservoir rock physics parameters. Specifically, the joint anisotropic acoustic-electrical measurement system is used to measure P- and S-wave velocities and complex electrical conductivity as functions of angle simultaneously on rock samples at various confining and pore pressure stages up to 4000 psi. The following is a list of parameters measured using the instrument:
- Compressional and shear wave velocities and their anisotropies
- Formation elastic properties and stiffness tensor
- Acoustic attenuation and attenuation tensor
- Complex electrical conductivity and conductivity tensor

Fluid Measurement System (FMS)

The fluid measurement system (FMS) is used to measure acoustic velocities in fluids at reservoir pressure and temperature conditions up to a maximum of 20,000 psi and 200° C. The FMS cell is equipped with a computer that provides full control and data acquisition of the FMS experiments. We use FMS to characterize reservoir fluid (brine, oil, and gas) systems, and to determine the effect of different gases (such as methane, N2, CO2 and pure hydrocarbon components) on reservoir oil behavior. The following is a partial list of important parameters which can be determined from FMS experiments:
- Fluid velocity under pressure change
- Fluid velocity under temperature change
- Fluid permeability

Impedance Analyzer – Keysight E4990A

This Impedance Analyzer is a complex resistivity measurement used for bench top measurements in a range of frequencies (20Hz to 120MHz). The analyzing probe can be used for samples with a maximum length of 11mm and variable diameters. The following is a partial list of important parameters which can be determined from these experiments:
- Conductivity
- Permittivity
- We use auxiliary instruments, for example Refractometer, to measure fluid conductivity if the sample analyzed is saturated.

Low Frequency Velocity Measurements

The technique used to determine elastic properties at low frequencies consists of a stress/strain system that deforms the rock at a frequency range of 1 to 2000 Hz. Measurements can be conducted at different confining and pore pressure stages to simulate reservoir conditions. Additionally we can control temperature of the pressure vessel (0 – 100 °C). Besides measuring low frequency velocities we are also able to perform ultrasonic (1 MHz) velocity measurements.

The following is a partial list of parameters that can be determined using the set up:
- Young’s modulus
- Poisson’s ratio
- Bulk modulus
- Shear modulus
- Compressional and shear wave velocities
- Attenuation
**Network Analyzer – Keysight ENA Series**

This Network Analyzer is a complex resistivity measurement used for bench top measurements in a range of frequencies (300 kHz to 20 GHz). The analyzing probe can be used for any sample; the only specification is the need for a flat polish surface, to allow full contact with the sample and reliable measurements. The following is a partial list of important parameters which can be determined from these experiments:

- Conductivity
- Permittivity

**The Spectral Induced Polarization (SIP) Resistivity Measuring Instrument**

The SIP resistivity measuring instrument measures the real and imaginary components of resistance for brine-saturated porous rocks in the frequency range of 1 mHz to 45 KHz.

The following is a partial list of the important parameters which can be determined from SIP experiments:

- Resistivity
- Tortuosity

**NUCLEAR LAB**

**FACILITY DETAILS**

The Nuclear lab is a part of the Center for Rock & Fluid Multiphysics. Nuclear lab capabilities include:

- Ultrasonic capabilities
- Pressure experiments
- Temperature experiments
- Fluid analysis
- Rock property analysis
- Image processing for digital rock physics

Tools include:

**Magritek® Low-Field 2-MHz NMR**

This Nuclear Magnetic Resonance (NMR) system operates a frequency of 2MHz and a magnetic field strength of 0.05T. For saturated porous media such as rocks and soils, the NMR response of this system is dependent on the size of the pore space as well as the hydrogen index of the saturating fluid. This non-destructive measurement leaves the core completely intact, while detecting hydrogen nuclei contained in the pore space through alternating magnetic fields. This allows for pore space properties to be determined without alteration of the core or pore space environment. Additional information about interstitial fluids and core mineralogy can be determined with some NMR methods. The following is a partial list of important parameters which can be determined from NMR experiments:

- Pore Size Distribution
- Porosity
- Permeability
- Fluid Viscosity
- Oil and Gas Compositions

These results are commonly used in conjunction with nitrogen adsorption and resistivity studies for a complete analysis of pore-space in both laboratory and downhole logging applications.
XRadia MicroXCT-400
The micro X-ray CT (computed tomography) machine provides high resolution 3D images of samples with up to 2" in diameter. The weight of the sample must be less than 15 kg. Image resolution of up to 1 μm can be achieved. Four different lenses allow us to cover a varying field of view size and a variable resolution. It is a non-destructive method to explore the internal structure of samples which might be unobservable with conventional 2D techniques, such as SEM.

The instrument chamber can house a pressure cell for studying in-situ change in internal microstructure with application of pore and confining pressures up to 2500 psi as well as a temperature controls between -5 °C to 50 °C. The following is a list of parameters that can be measured using the instrument:
- Rock microstructure for oil and gas exploration
- In situ measurement during imaging (e.g. ultrasonic velocities)
- Semiconductor packaging development and failure analysis
- Life-science research
- Advanced material characterization

The lab also includes a supercomputer and full waveform inversion acoustic set-up.

POROELASTIC LAB

The Poroelastic lab is part of the Center for Rock & Fluid Multiphysics. Instrumentation enables poroelastic property measurements and ultrasonic velocity measurements.

The lab’s primary tool is the NER AutoLab 1500. AutoLab 1500 is a servo-hydraulic operated system for biaxial measurements with software-controlled arbitrary stress paths on rock specimens up to 50.8 mm (2.0 in) in diameter at in-situ stress conditions with pore pressure and temperature controls. The high pressure biaxial system consists of a pressure vessel with an internal piston for differential stress and servo-hydraulic intensifiers for differential stress, confining and pore pressure. This instrument allows us to make measurements at reservoir pressures up to 69 MPa (10,000 psi) and temperatures up to 120°C (248°F).

The instrument’s key features are the following:
- Servo-hydraulic control of confining pressure, pore pressure, flow rate, strain rate, and force.
- Control of stresses and temperatures at reservoir conditions.
- Pore pressure intensifier compatible with water, brine, oil, and gas (including CO2).
- AutoLab software for system data acquisition and reduction.
- Integrated electronics console for servo-amplifiers and signal conditioning.
- The NER Autolab 1500 measures the following parameters:
  - Compressional and shear wave velocities
  - Electrical resistivity
  - Permeability
X-RAY DIFFRACTION & COMPUTED TOMOGRAPHY

MALVERN PANALYTICAL EMPYREAN X-RAY DIFRACTOMETER
The Malvern Panalytical Empyrean X-ray diffractometer provides crystallographic and compositional information critical to understanding part mechanical performance. It features small- and wide-angle X-ray scattering (SAXS/WAXS) and has the ability to test samples at temperatures ranging from −200 °C to 1100 °C. It can also capture information on texture, residual stress and pair distribution functions.
- Cu and Mo radiation for low- and high-energy X-ray diffraction (XRD)
- Reflection/transmission geometry with rotating sample for improved statistics
- Spinning capillary
- Bragg–Brentano optics and focusing geometry
- Residual strain measurements
- Cu/Mo focusing mirror and monocapillary 100 µm beam for microdiffraction
- 5-axis Eulerian cradle for texture mapping
- SAXS/WAXS
- Grazing incidence XRD
- Pair distribution function
- Galipix 3D area detector
- In situ stages from −200 to 1100 °C (−328 to 2012 °F)

PANALYTICAL PW3040 X-RAY DIFRACTOMETER
This PANalytical (nee Philips) instrument is equipped with a versatile computer-controlled diffractometer and is ideal for routine XRD analysis of powder/polycrystalline specimens. Collection and search software includes the current ICDD database.
- Routine powder/polycrystalline diffraction
- Computer Controlled Stage with X’pert Pro MPD
- Data Collector and DataView Software
- HighScore Search Software
- Current ICDD Database
- Cu and Cr Tubes Available

ZEISS VERSA X-RAY MICROSCOPE
The Zeiss Xradia Versa 3D X-ray microscope enables cutting-edge, nondestructive tomographic imaging and grain reconstruction. X-ray tomography (µ-XCT) allows for the collection of both surface and internal renderings, which are used to distinguish between phases and identify defects such as porosity. Nondestructive diffraction contrast tomography (DCT) provides direct 3D crystallographic grain reconstructions for crystalline materials. Our lab offers the unique ability to first nondestructively characterize pore distributions in sample parts using the Zeiss Xradia Versa, then to mechanically test those same parts using the load frames described below to correlate defect structures and mechanical behavior.
- Absorption contrast tomography: resolution to 0.7 µm; up to 160 kV to probe metallic samples up to 4 mm in diameter; automated center shift and reconstruction; automated ring artifact elimination
- Phase contrast tomography: improved resolution at phase boundaries to distinguish particle dispersions
- Dual-energy tomography: low- and high-energy scans allow for segmentation based on differential X-ray absorption
- Diffraction contrast tomography: grain mapping provides crystallographic orientation of samples; grain size approximation enables the preparation of samples for synchrotron experiments
X-RAY PHOTOELECTRON SPECTROSCOPY

ROCKY MOUNTAIN ENVIRONMENTAL XPS USER FACILITY
The Rocky Mountain Environmental X-ray Photoelectron Spectroscopy (E-XPS) center is a user facility for state-of-the-art surface analysis measurements.

XPS measures material surface compositions by measuring the intensity of photoelectrons emitted as a function of the incident X-ray energy. Spectra features are used to identify the surface species present and calculate the fraction of the surface occupied by each.

The Rocky Mountain E-XPS extends these traditional capabilities by enabling measurements in a range of environmental conditions.

The instrument, based on the HiPP lab system from Scienta Omicron, can measure XPS at various pressures and temperatures, and with variable chemical compositions.

Unlike traditional UHV XPS systems, the Rocky Mountain E-XPS can therefore look at surface states for functional materials in relevant environments and with optional electronic biasing for analysis of non-equilibrated surface states.

SOURCE
The HiPP Lab from Scienta Omicron utilizes a high power, high energy resolution monochromated Al-Kα source with 600 W continuous operation and modified for near-ambient pressure XPS measurements.

ANALYZER
The energy analyzer boasts the following specifications:

- Energy resolution < 15 meV FWHM at 20 eV pass energy and 500 eV kinetic energy
- Pass energies of 20, 50, 100, 200, and 500 eV.
- The analyzer can operate in standard transmission mode, or collect one-shot measurements to analyze spectra as a function of X-ray energy and either angle (angular mode) or lateral position (spatial mode).
- Transmission mode kinetic energy range: 20 eV – 1500 eV
- Angular mode kinetic energy range: 100 eV – 1500 eV. Parallel angular range: ± 9 degrees.
- Spatial mode kinetic energy range: 20 eV – 1500 eV. Spatial resolution: < 10 μm

SAMPLE ENVIRONMENT CONDITIONS
- Gas pressures of up to 30 mbar
- Temperatures of up to 700 C.
- Gas flow management system allows mixing of two separate feed gases, controlled via mass flow controllers, plus optional water vapor.
- Chamber exhaust composition quantified and monitored via quadrupole mass spectrometer.
- Optional electrical leads for operando biasing of samples.